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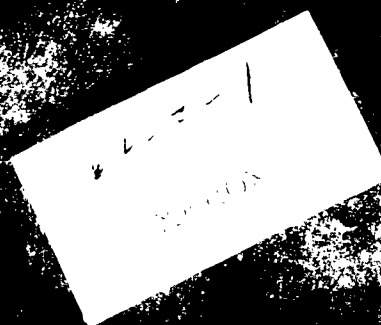


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NORTHROP CORPORATION

NORAIR DIVISION



NORTHROP AIRCRAFT, INC.



NORTHROP DIVISION

REPORT NO. NOR-60-40

EVALUATION OF UNICHROME CF-500 CRACK-FREE
CHROMIUM ELECTROPLATING PROCESS

8 February 1960

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REVISIONS

| CHG. NO. | DATE | ENGR. | PAGES AFFECTED | REMARKS |
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1. OBJECT

To compare the characteristics of Unichrome CF-500 ("crack-free" chromium) to conventional electrodeposits by: comparing the corrosion and wear resistance of deposits produced by each process; determining what effect each deposit has on the fatigue strength and embrittlement behavior of high strength steel; and observing the stress condition and deposit cracking in electrodeposits produced by each process.

2. CONCLUSIONS

The corrosion and wear resistance characteristics of Unichrome CF-500 electrodeposits, when used as a protective coating for steel, are superior to conventional chromium electrodeposits. The conventional chromium electroplating process, however, produces deposits which result in longer fatigue life when applied to high-strength steel, induce less hydrogen embrittlement, facilitate better post-plate embrittlement relief, and contain less tensile stress. Electrodeposits from the Unichrome CF-500 electrolyte were not entirely crack-free, but there was a significant reduction in the number of cracks as compared to those observed in the conventional chromium electrodeposit.

3. PROCEDURE AND RESULTS

3.1 Electroplating Processes

The electroplating of chromium was performed in laboratory plating baths of 36 gallon capacity according to the procedures outlined in the appendix.

3.2 Corrosion Protection

Steel test specimens (normally used for bend tests) 1/4 inch in diameter by 4 inches in length were electroplated by each process to thicknesses of 0.25, 0.50, 0.75 and 1.0 mil (4 specimens for each thickness) and subjected to a 240 hour salt spray test as prescribed in Method 811.1 (20 per cent NaCl) of Federal Test Method Standard No. 151a. The results of these tests, as shown in Figure 1, proved that Unichrome CF-500 chromium electrodeposits are superior for the corrosion protection of steel.

3.3 Wear Resistance

Four beryllium copper panels 0.040 by 4 by 4 inches were electroplated to a thickness of approximately 0.15 mil by each process. The panels were weighed, subjected to 500 revolutions on a Taber Abraser using a 1000 gram load and 17-G wheels, and reweighed to determine weight loss of each panel. New abrasive wheels were used for each series of tests. The results of these tests, given in Table I, indicated an average weight loss of 661 milligrams.

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for Unichrome CF-500 deposits and 1039 milligrams for the conventional chromium electrodeposits.

3.4 Fatigue Strength

3.4.1 Fatigue test specimens, machined to the configuration indicated in Figure 2, were shot peened in accordance with Specification MIL-S-13165A, electroplated by each process to thicknesses of 0.25, 0.50, and 1.0 mil, and subjected to high-speed alternating stresses on an R. R. Moore fatigue testing machine until failure. The material used for the fatigue test specimens, which was procured from the Earle M. Jorgensen Company, was certified as follows:

Description and Specifications

Size

E-4340 CF ANN AQ
MIL-S-5000A COND C-4

1/2 inch RD

Chemical Analysis

| Heat No. | Mill | C | Mn | P | S | Si | Ni | Cr | Mo | G/S |
|----------|------|-----|-----|------|------|-----|------|-----|-----|------|
| 13232 | B&L | .39 | .74 | .016 | .011 | .25 | 1.77 | .81 | .27 | Fine |

3.4.2 In the testing machine the specimens functioned as a simple beam symmetrically loaded at two points. As the beam was rotated the stresses originally above the neutral axis of the specimen were first reversed from compression to tension and, upon completion of one revolution, were again reversed. During one complete revolution the specimen passed through a complete cycle of flexural stresses. Stress levels of 95,000, 100,000, and 105,000 psi were used in testing each type of deposit at each thickness range. The results of these tests, given in Figures 3, 4, and 5 indicate a slight loss in fatigue strength when Unichrome CF-500 electrodeposits are used.

3.5 Embrittlement Characteristics

Steel bend test specimens 1/4 inch in diameter by 4 inches in length were electroplated by each process to thicknesses of 0.25, 0.50, 0.75 and 1.0 mil (4 specimens for each thickness and for each process) and bent to fracture on a Northrop bend test apparatus. One half of the specimens in each group were baked at 375 F \pm 25 for 3 hours immediately after plating, and before bending, for relief of hydrogen embrittlement. The rest of the specimens were

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bent to fracture immediately after electroplating without benefit of relief treatment. The comparison of fracture angle, given in Figure 6 (each bar is the average 4 specimens), indicates that the Unichrome CF-500 process exhibits more embrittlement and, further, the embrittlement induced is difficult to dispel by post-plate baking.

3.6 Stress Condition of Deposits

A Brenner-Senderoff Spiral Contractometer (see Figure 7 and reference 5.4) an instrument recently developed for measurement of stresses in electrodeposits, was used to compare the two processes for total amount of tensile stress present in each deposit at a given thickness. The results of stress measurements on each deposit, presented in Figure 8, indicate that more tensile stress is present in the Unichrome CF-500 electrodeposit than exists in the conventional electrodeposit of the same thickness. It should be noted that the stress which was determined at given thicknesses is an average stress and not a suitable quantity for studying the change of stress with thickness of deposit.

3.7 Deposit Cracking

Four bend test specimens were electroplated by each process to a thickness of from 0.5 to 0.75 mil and one from each process was examined metallographically to determine the extent of deposit cracking. Cracks in the chromium electrodeposit can be seen in the photo-micrographs in Figure 9. The picture on the right, in which at least eight cracks are visible, is an electrodeposit produced from a conventional process while the picture on the left, showing but a single crack, is an electrodeposit produced from the Unichrome CF-500 process. Thus, it was indicated the Unichrome CF-500 process does significantly reduce the number of cracks but, it should be noted, such deposits are not entirely crack-free.

4. DISCUSSION

- 4.1 The Unichrome CF-500 deposit offers better corrosion protection than conventional chromium, ostensibly because there are fewer corrosion-admitting cracks in the deposit. The absence of an extensive network of cracks, in addition to facilitating better corrosion protection, should make the Unichrome CF-500 deposit useful in oxidation protection systems for molybdenum (at high temperature) which depend on composite coatings of chromium and nickel. Past failures of these composite coatings, in most cases, have been attributed to cracks in the chromium deposit which allow the

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molybdenum to diffuse and form compounds (nickel molybdate) which have a tendency to spall.

- 4.2 The Unichrome CF-500 deposit is surprisingly more wear resistant than conventional deposits but care should be exercised in application of such deposits for wear resistance because of the embrittlement problem.
- 4.3 The Unichrome CF-500 process produces more hydrogen embrittlement and does not respond to post-plate baking as well as conventional processes. The theory that the hydrogen gas escapes through cracks or grain boundaries is further substantiated by the fact that less embrittlement is encountered with conventional chromium deposits showing extensive crack patterns than with Unichrome CF-500 deposits in which cracks are relatively few.
- 4.4 The presence of high residual tensile stresses during chromium deposition from the conventional processes and the resultant formation of cracks is also believed to cause a reduction in the fatigue strength of the basis metal. The postulation upon which this work was justified was that perhaps the absence of cracks in the so-called "crack-free" chromium deposit was due to less residual tensile stresses being present, and the absence of such stresses would greatly enhance the fatigue strength of the basis metal. The opposite proved to be the case. Using the spiral contractometer it was found that the "crack-free" deposits were actually under very high tensile stress. The conventional chromium deposits were under lower tensile stress, probably because the deposit was stress relieved by deposit cracking.
- 4.5 It can be concluded that the absence of deposit cracking in the Unichrome CF-500 deposit is due to the fact that such deposits have greater tensile strength, probably because of lower oxide inclusions, than the tensile stresses which build up in the deposit.

5. REFERENCES

- 5.1 Federal Test Method Standard No. 151a, "Metals; Test Methods", 6 May 1959.
- 5.2 Federal Specification QQ-C-320, "Chromium Plating (Electrodeposited)", 3 July 1957.
- 5.3 Military Specification MIL-S-13165A, "Shot Peening of Ferrous Metal Parts", 26 March 1956.

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- 5.4 Brenner, Abner, and Senderoff, Seymour "A Spiral Contractometer for Measuring Stress in Electrodeposits", United States Department of Commerce National Bureau of Standards Research Paper RP 1953, Vol. 42, February 1949.
- 5.5 Cohen, Bennie "Effect of Shot Peening Prior to Chromium Plating on the Fatigue Strength of High Strength Steel", paper presented at AES 45th Annual Proceedings, Cincinnati, Ohio, May 1958.
- 5.6 Kushner, J. B. "Stress in Electrodeposited Coatings", METAL FINISHING, May 1956.
- 5.7 Seyb, E. J. and Rowan, W. H. "Thicker Decorative Chromium for Better Corrosion Resistance", PLATING, February 1959.
- 5.8 Bulletin CF-500, "Installation, Operation, and Control of the Unichrome Crack-Free Chromium Plating Solution CF-500", Metal and Thermit Corporation, May 1959.
- 5.9 Bulletin 4200 "Operating Instructions for R. R. Moore High Speed Fatigue Testing Machine", Baldwin-Lima-Hamilton Corporation, 1952.

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TABLE I. WEIGHT LOSS IN TABER ABRASER TESTS

| Specimen No. | First Weight (g) | Second Weight (g) | Weight Loss (mg) |
|-----------------------|------------------|-------------------|------------------|
| Unichrome CF-500 | | | |
| 1 | 75.7060 | 75.6193 | 867 |
| 2 | 76.1600 | 76.0610 | 690 |
| 3 | 76.0575 | 75.9929 | 546 |
| 4 | 75.6636 | 75.6093 | 543 |
| | | | 661 Avg. |
| Conventional Chromium | | | |
| 5 | 75.4760 | 75.3640 | 1120 |
| 6 | 75.7055 | 75.5945 | 1110 |
| 7 | 75.0813 | 74.9830 | 983 |
| 8 | 75.3005 | 75.2064 | 941 |
| | | | 1039 Avg. |

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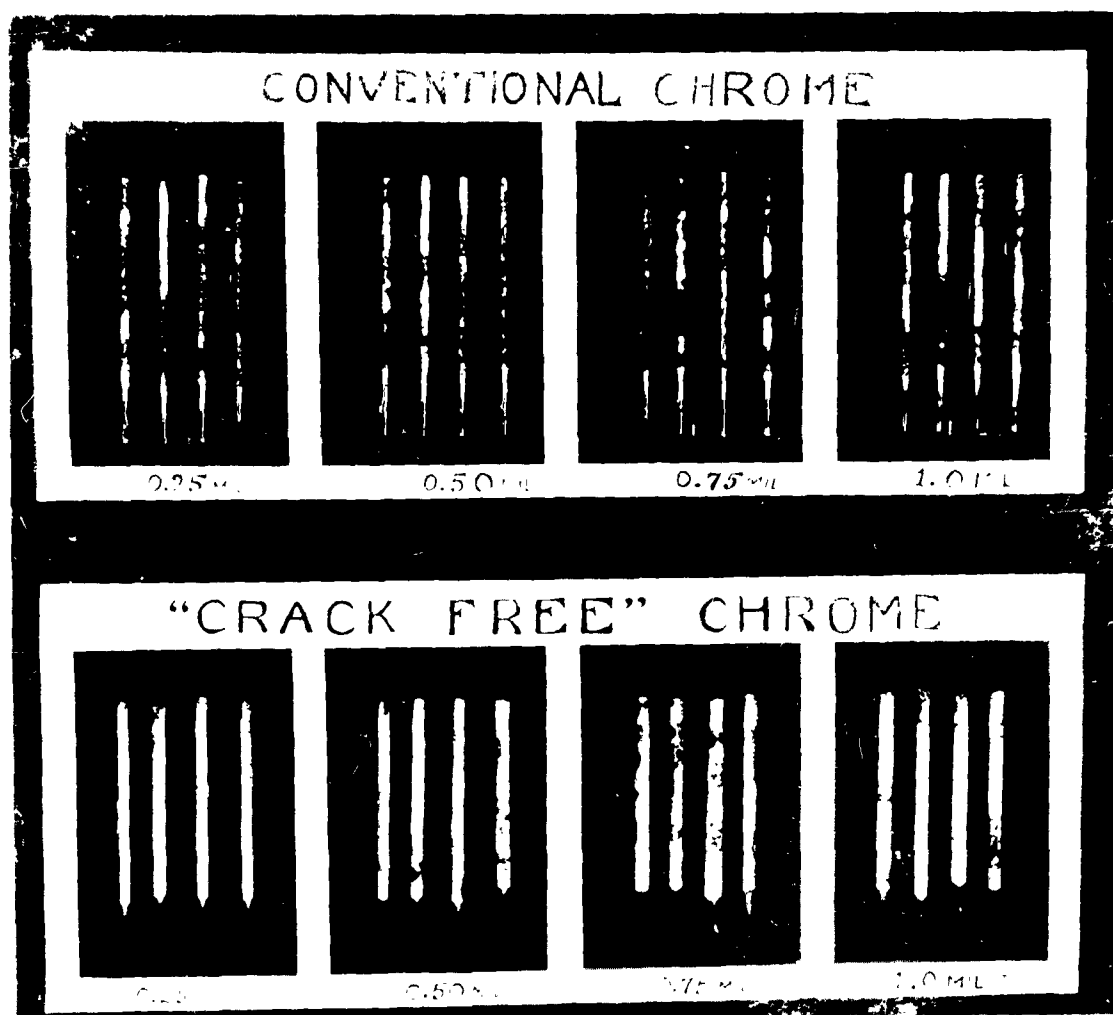


Figure 1. SAE 1095 Steel Specimens after Chromium Plating with Crack-Free (Unichrome CF-500) and Conventional Chromium and 240 Hours Exposure in the Salt Spray

Figure 2. Fatigue Test Specimen, in Inches

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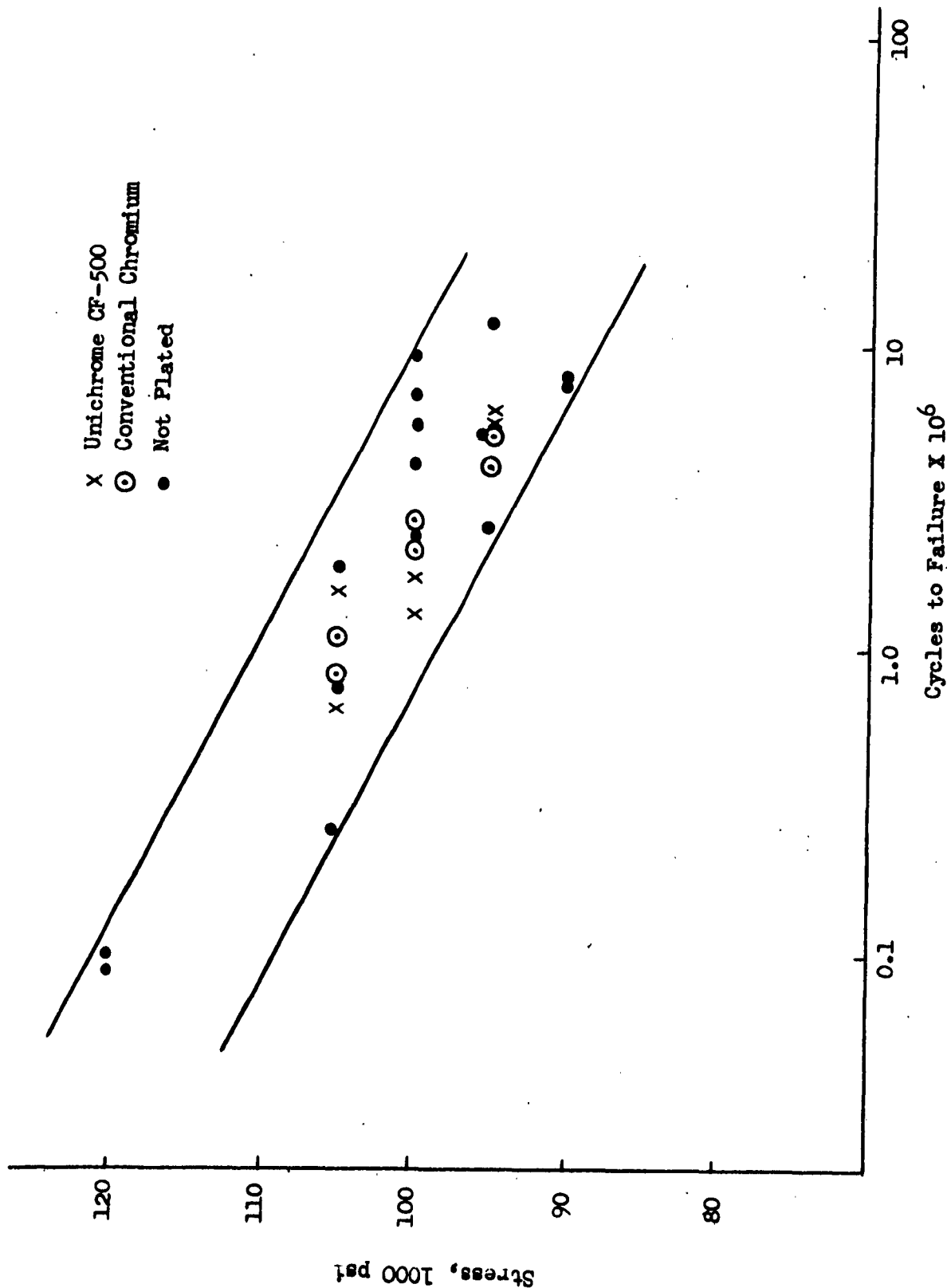
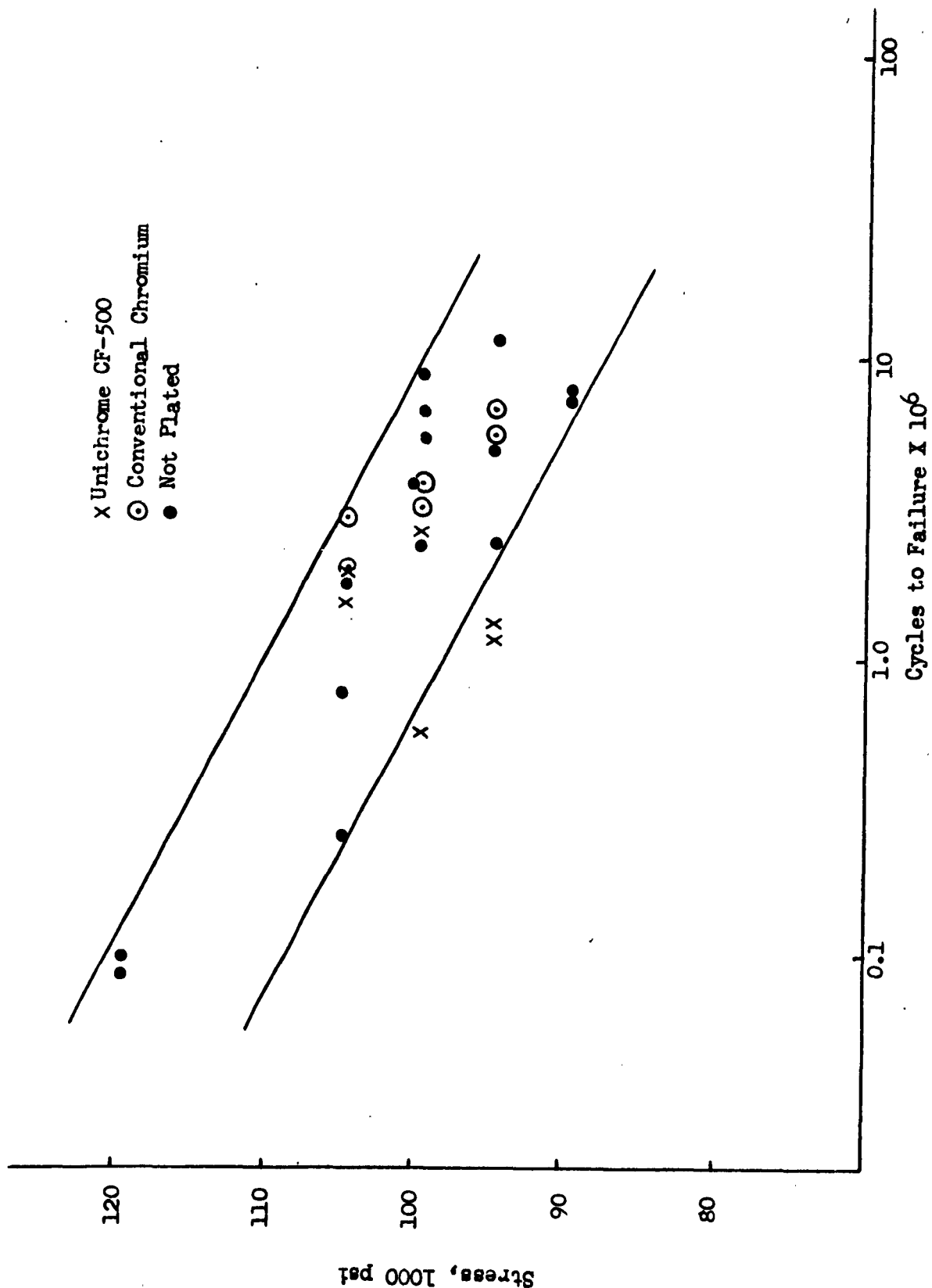
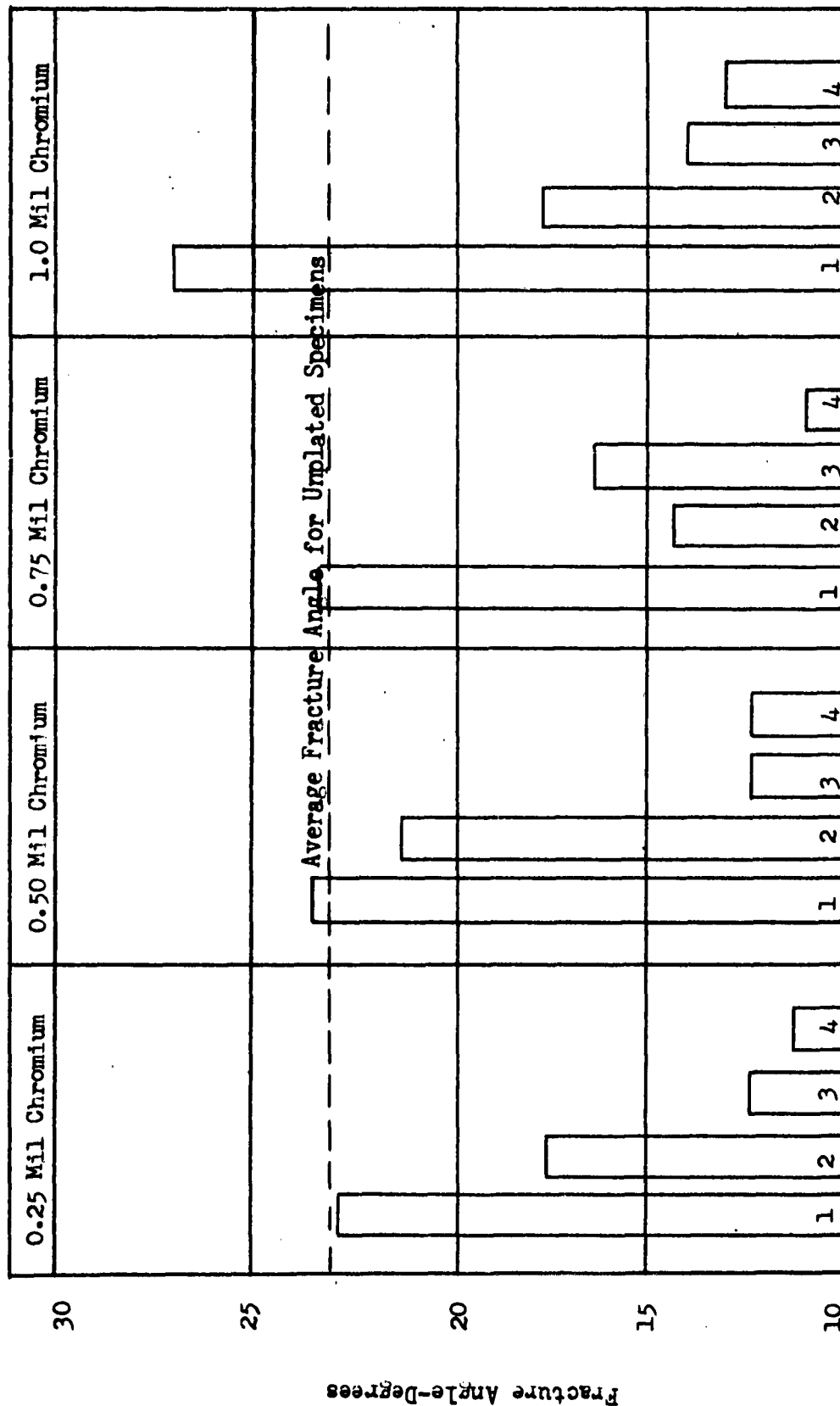


Figure 4. Cycles to Failure vs. Alternating Stress; 0.0005 inch thick Chromium Deposit; AISI 4340, Heat Treat 260-280 ksi, Shot Peened

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- 1 - Conventional Chromium: Post-Plate Baked at 375 F ± 25 for 3 Hours
- 2 - Unichrome CF-500 Chromium: Post-Plate Baked at 375 F ± 25 for 3 Hours
- 3 - Conventional Chromium: Not Baked
- 4 - Unichrome CF-500: Not Baked

Figure 6. Bend Test Results - Embrittlement Characteristics of Conventional and Unichrome CF-500 Chromium Electrodeposits

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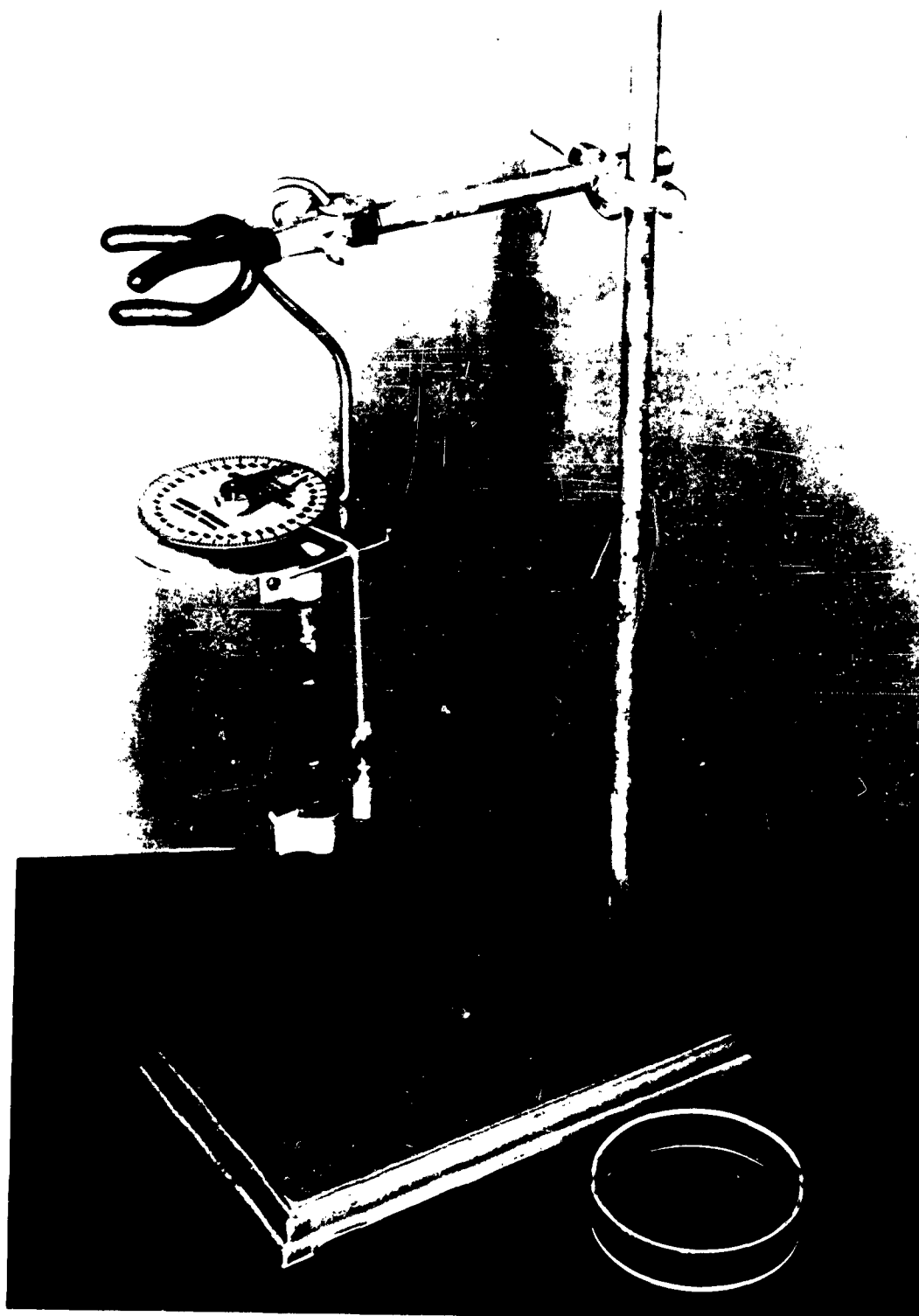


Figure 7. Brenner-Senderoff Spiral Contractometer in Position for Calibration Against a 1-ounce Weight

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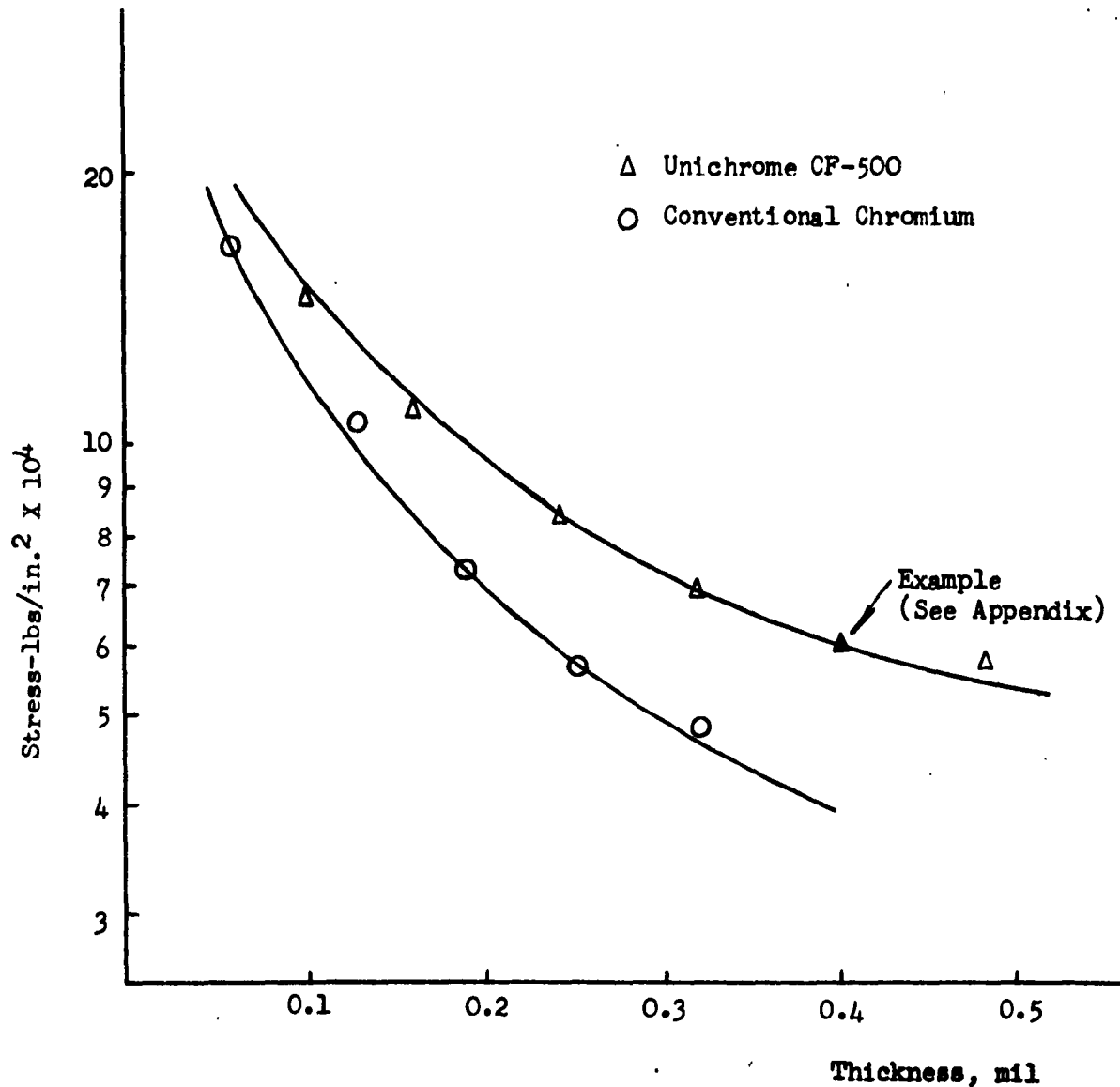


Figure 8. Residual Stress Curves for Unichrome CF-500 and Conventional Chromium Deposits

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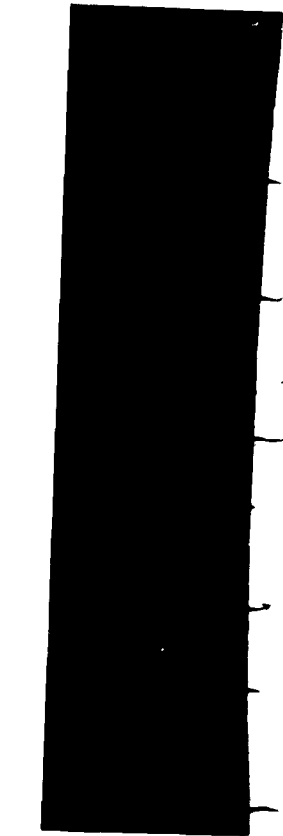
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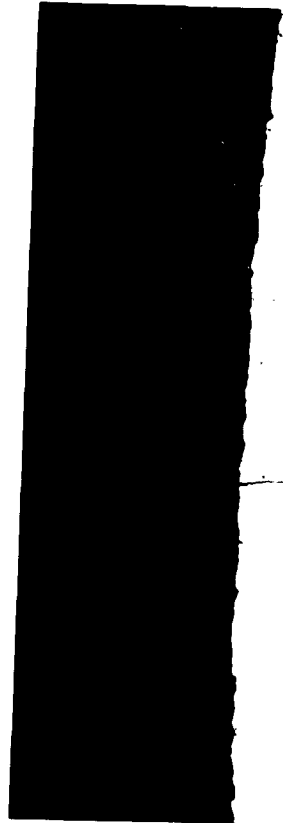
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Mag. 250X

Unichrome CF-500 Chromium Deposit



Mag. 250X

Conventional Chromium Deposit

Figure 9. Deposit Cracking in Unichrome CF-500 and Conventional Chromium Electrodeposits

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APPENDIX

SOLUTION COMPOSITION AND OPERATING CONDITIONS, PROCESSING SEQUENCE, AND CALCULATIONS

1. Proprietary Products

Northwest Cleaner #470 - Alert Supply Company
Unichrome CF-500 - Metal and Thermit Corporation

2. Solution Composition and Operating Conditions

Electrocleaner

| | |
|------------------------|---------------|
| Northwest cleaner #470 | 8.0 oz/gal. |
| Temperature | 145 to 155 F |
| Current density | 50 to 100 asf |
| Volts (anodic) | 6 to 8 |

Acid Dip

| | |
|-------------------|---------------|
| Hydrochloric acid | 10% by weight |
| Temperature | room |

Crack-Free Chromium Bath

| | |
|------------------|----------------|
| Unichrome CF-500 | 36.5 oz/gal. |
| Distilled water | balance |
| Temperature | 148 to 152 F |
| Current density | 288 to 432 asf |
| Agitation | plant air |

Conventional Chromium Bath

| | |
|-----------------|----------------|
| Chromic acid | 33.0 oz/gal. |
| Sulfate | 0.33 oz/gal. |
| Distilled water | balance |
| Temperature | 110 to 120 F |
| Agitation | plant air |
| Current density | 100 to 200 asf |

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3. Processing Sequence

1. Shot peen per MIL-S-13165A
2. Electroclean for 3 minutes
3. Rinse in cold water
4. Acid dip for 10 seconds
5. Cold water rinse (thoroughly)
6. Etch with reverse current (6 volts) for 30 seconds in the regular plating bath
7. Chromium plate per Federal Specification QQ-C-320 in Unichrome CF-500 or in a conventional chromium plating bath to the desired thickness
8. Cold water rinse
9. Hot water rinse

Note: Steps 4 and 6 were used in this work for the purpose of obtaining better adhesion. Such practice is not advisable for production processes, however, since there is a danger of introducing harmful contaminants (iron and chlorine) into the plating bath.

4. Calculations - Brenner-Senderoff Spiral Contractometer

- 4.1 The formulas by which stress was determined in the electrodeposits are based on those of Stoney with modifications by Brenner to adapt them for use with a helix instead of a straight strip. The symbols used are defined as follows:

- S = stress
- E = Young's modulus of the basis metal strip
- p = pitch of the strip
- t = thickness of basis metal strip
- d = thickness of chromium deposit
- $\Delta(\frac{1}{r})$ = change of curvature induced by the deposit
- h = height of plated portion of helix
- D = angular deflection of helix in radians
- D = angular deflection of dial needle in degrees
- C = outside diameter of helix
- K = deflection constant of helix

The basic formula is:

$$S = \frac{Et^2}{6d} \times \Delta(\frac{1}{r}) \quad (1)$$

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The change of curvature caused by deposition of chromium is:

$$\Delta\left(\frac{1}{r}\right) = \frac{D_p}{\pi Ch} \quad (2)$$

Substituting in equation (1) and rearranging terms, the formula for calculating stress using a helix becomes:

$$s = \frac{Et^2 p}{6\pi Ch} \times \frac{D}{d} \quad (3)$$

Young's modulus of the helix may be calculated with the following formula using the value of K obtained by calibrating the helix against a 1 ounce weight:

$$E = \frac{2.16 \times 10^4 KCh}{p^2 t^3} \quad (4)$$

E is in lb/in.² when C, h, p, and t are in inches.

Substituting in equation (3) the working formula is:

$$s = \frac{2.16 \times 10^4 K}{6\pi pt} \times \frac{D}{d} \quad (5)$$

Example

In the stress measurement indicated in Figure 8, the following dimensions of the helix were accurately measured:

| | |
|-------------------------------------------|-------------------------|
| W = deflection produced by 1-ounce weight | 188 degrees |
| C = outside diameter of helix | 0.756 inches |
| t = thickness of basis metal strip | 0.013 inches |
| p = pitch of the helix | 0.756 inches |
| h = height of plated portion | 4.500 inches |
| \bar{D} = angular deflection of dial | 372 degrees |
| K = deflection constant = $\frac{1}{16W}$ | $= 3.32 \times 10^{-4}$ |

\bar{D} is converted to deflection of the helix in radians, D:

$372^\circ/10 = 37.2$ degrees deflection of helix, since the gear ratio was 10 to 1.

$$37.2 \times \pi/180 = 0.649 \text{ radians} = D$$

The thickness (0.0004 inch) of the chromium was calculated from the weight gain of the helix using the following formula:

| | | |
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$$d = \frac{g}{G \times 16.4A}$$

where d = thickness
g = weight of deposit
in grams
G = density in grams/cc
A = area of deposit in in.²
16.4 = conversion factor

The above values were used to determine the stress employing equation (5) as follows:

$$S = \frac{2.16 \times 10^4 K}{6 \pi \text{ pt}} \times \frac{D}{d} = \frac{2.16 \times 3.32 \times 0.649}{6 \pi \times 0.756 \times 0.013 \times 4 \times 10^{-4}} = 62.8 \times 10^3 \text{ psi}$$